

checked by IT
on 9/6/17

CERTIFICATION

SDG No: JC25442 Laboratory: Accutest, New Jersey
Site: BMSMC, Building 5 Area, PR Matrix: Groundwater

SUMMARY: Groundwater samples (Table 1) were collected on the BMSMC facility – BMSMC, Building 5 Area, PR. The BMSMC facility is located in Humacao, PR. Samples were taken July 22-26, 2016 and were analyzed in Accutest Laboratory of Dayton, New Jersey for 1,4-Dioxane and Naphthalene. The results were reported under SDG No.: JC25442. Results were validated using the latest validation guidelines (July, 2015) of the EPA Hazardous Waste Support Section. The analyses performed are shown in Table 1. Individual data review worksheets are enclosed for each target analyte group. The data sample organic data samples summary form shows for analytes results that were qualified.

In summary the results are valid and can be used for decision taking purposes.

Table 1. Samples analyzed and analysis performed

| SAMPLE ID | SAMPLE DESCRIPTION | MATRIX | ANALYSIS PERFORMED |
|------------|--------------------|-------------|------------------------------------|
| JC25442-1 | OSMW-4D | Groundwater | 1,-4-dioxane and Naphthalene (SIM) |
| JC25442-1 | OSMW-4D | Groundwater | 1,-4-dioxane (SCAN) |
| JC25442-2 | OSMW-4S | Groundwater | 1,-4-dioxane and Naphthalene (SIM) |
| JC25442-2 | OSMW-4S | Groundwater | 1,-4-dioxane (SCAN) |
| JC25442-2D | OSMW-4S MSD | Groundwater | 1,-4-dioxane and Naphthalene (SIM) |
| JC25442-2S | OSMW-4S MS | Groundwater | 1,-4-dioxane and Naphthalene (SIM) |
| JC25442-3 | OSMW-5D | Groundwater | 1,-4-dioxane and Naphthalene (SIM) |
| JC25442-3 | OSMW-5D | Groundwater | 1,-4-dioxane (SCAN) |
| JC25442-4 | OSMW-5S | Groundwater | 1,-4-dioxane and Naphthalene (SIM) |
| JC25442-4 | OSMW-5S | Groundwater | 1,-4-dioxane (SCAN) |

Reviewer Name: Rafael Infante
Chemist License 1888

Signature:
Date:

Rafael Infante
August 16, 2016



SGS Accutest

Report of Analysis

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| | | | |
|--------------------------|--------------------------------|------------------------|----------|
| Client Sample ID: | OSMW-4D | Date Sampled: | 08/05/16 |
| Lab Sample ID: | JC25442-1 | Date Received: | 08/06/16 |
| Matrix: | AQ - Ground Water | Percent Solids: | n/a |
| Method: | SW846 8270D BY SIM SW846 3510C | | |
| Project: | BMSMC, Building 5 Area, PR | | |

| | File ID | DF | Analyzed | By | Prep Date | Prep Batch | Analytical Batch |
|--------|-----------|----|----------|----|-----------|------------|------------------|
| Run #1 | F159723.D | 1 | 08/10/16 | AD | 08/09/16 | OP96175A | EF6717 |
| Run #2 | 4M67260.D | 1 | 08/10/16 | JJ | 08/09/16 | OP96175A | E4M3041 |

| | Initial Volume | Final Volume |
|--------|----------------|--------------|
| Run #1 | 1000 ml | 1.0 ml |
| Run #2 | 1000 ml | 1.0 ml |

| CAS No. | Compound | Result | RL | MDL | Units | Q |
|----------|-------------|-----------------|------|-------|-------|---|
| 91-20-3 | Naphthalene | ND ^a | 0.10 | 0.029 | ug/l | |
| 123-91-1 | 1,4-Dioxane | 28.4 | 1.0 | 0.049 | ug/l | |

| CAS No. | Surrogate Recoveries | Run# 1 | Run# 2 | Limits |
|-----------|----------------------|--------|--------|---------|
| 4165-60-0 | Nitrobenzene-d5 | 66% | 62% | 24-125% |
| 321-60-8 | 2-Fluorobiphenyl | 66% | 52% | 19-127% |
| 1718-51-0 | Terphenyl-d14 | 76% | 89% | 10-119% |

(a) Result is from Run# 2



ND = Not detected MDL = Method Detection Limit
 RL = Reporting Limit
 E = Indicates value exceeds calibration range

J = Indicates an estimated value
 B = Indicates analyte found in associated method blank
 N = Indicates presumptive evidence of a compound

SGS Accutest

Report of Analysis

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| | | | |
|--------------------------|--------------------------------|------------------------|----------|
| Client Sample ID: | OSMW-4S | Date Sampled: | 08/05/16 |
| Lab Sample ID: | JC25442-2 | Date Received: | 08/06/16 |
| Matrix: | AQ - Ground Water | Percent Solids: | n/a |
| Method: | SW846 8270D BY SIM SW846 3510C | | |
| Project: | BMSMC, Building 5 Area, PR | | |

| | File ID | DF | Analyzed | By | Prep Date | Prep Batch | Analytical Batch |
|--------|-----------|----|----------|----|-----------|------------|------------------|
| Run #1 | F159724.D | 1 | 08/10/16 | AD | 08/09/16 | OP96175A | EF6717 |
| Run #2 | 4M67273.D | 1 | 08/10/16 | JJ | 08/09/16 | OP96175A | E4M3041 |

| | Initial Volume | Final Volume |
|--------|----------------|--------------|
| Run #1 | 970 ml | 1.0 ml |
| Run #2 | 970 ml | 1.0 ml |

| CAS No. | Compound | Result | RL | MDL | Units | Q |
|----------|-------------|-----------------|------|-------|-------|---|
| 91-20-3 | Naphthalene | ND ^a | 0.10 | 0.030 | ug/l | |
| 123-91-1 | 1,4-Dioxane | 49.8 | 1.0 | 0.050 | ug/l | |

| CAS No. | Surrogate Recoveries | Run# 1 | Run# 2 | Limits |
|-----------|----------------------|--------|--------|---------|
| 4165-60-0 | Nitrobenzene-d5 | 73% | 65% | 24-125% |
| 321-60-8 | 2-Fluorobiphenyl | 69% | 55% | 19-127% |
| 1718-51-0 | Terphenyl-d14 | 76% | 91% | 10-119% |

(a) Result is from Run# 2



ND = Not detected MDL = Method Detection Limit
 RL = Reporting Limit
 E = Indicates value exceeds calibration range

J = Indicates an estimated value
 B = Indicates analyte found in associated method blank
 N = Indicates presumptive evidence of a compound

SGS Accutest

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| | | | |
|--------------------------|--------------------------------|------------------------|----------|
| Client Sample ID: | OSMW-5D | Date Sampled: | 08/05/16 |
| Lab Sample ID: | JC25442-3 | Date Received: | 08/06/16 |
| Matrix: | AQ - Ground Water | Percent Solids: | n/a |
| Method: | SW846 8270D BY SIM SW846 3510C | | |
| Project: | BMSMC, Building 5 Area, PR | | |

| | File ID | DF | Analyzed | By | Prep Date | Prep Batch | Analytical Batch |
|--------|-----------|----|----------|----|-----------|------------|------------------|
| Run #1 | F159725.D | 1 | 08/10/16 | AD | 08/09/16 | OP96175A | EF6717 |
| Run #2 | 4M67262.D | 1 | 08/10/16 | JJ | 08/09/16 | OP96175A | E4M3041 |

| | Initial Volume | Final Volume |
|--------|----------------|--------------|
| Run #1 | 1000 ml | 1.0 ml |
| Run #2 | 1000 ml | 1.0 ml |

| CAS No. | Compound | Result | RL | MDL | Units | Q |
|----------|-------------|-----------------|------|-------|-------|---|
| 91-20-3 | Naphthalene | ND ^a | 0.10 | 0.029 | ug/l | |
| 123-91-1 | 1,4-Dioxane | 55.2 | 1.0 | 0.049 | ug/l | |

| CAS No. | Surrogate Recoveries | Run# 1 | Run# 2 | Limits |
|-----------|----------------------|--------|--------|---------|
| 4165-60-0 | Nitrobenzene-d5 | 64% | 54% | 24-125% |
| 321-60-8 | 2-Fluorobiphenyl | 59% | 45% | 19-127% |
| 1718-51-0 | Terphenyl-d14 | 63% | 73% | 10-119% |

(a) Result is from Run# 2



ND = Not detected MDL = Method Detection Limit
 RL = Reporting Limit
 E = Indicates value exceeds calibration range

J = Indicates an estimated value
 B = Indicates analyte found in associated method blank
 N = Indicates presumptive evidence of a compound

SGS Accutest

Report of Analysis

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| | | | |
|--------------------------|--------------------------------|------------------------|----------|
| Client Sample ID: | OSMW-5S | Date Sampled: | 08/05/16 |
| Lab Sample ID: | JC25442-4 | Date Received: | 08/06/16 |
| Matrix: | AQ - Ground Water | Percent Solids: | n/a |
| Method: | SW846 8270D BY SIM SW846 3510C | | |
| Project: | BMSMC, Building 5 Area, PR | | |

| | File ID | DF | Analyzed | By | Prep Date | Prep Batch | Analytical Batch |
|--------|-----------|----|----------|----|-----------|------------|------------------|
| Run #1 | F159726.D | 1 | 08/10/16 | AD | 08/09/16 | OP96175A | EF6717 |
| Run #2 | 4M67263.D | 1 | 08/10/16 | JJ | 08/09/16 | OP96175A | E4M3041 |

| | Initial Volume | Final Volume |
|--------|----------------|--------------|
| Run #1 | 980 ml | 1.0 ml |
| Run #2 | 980 ml | 1.0 ml |

| CAS No. | Compound | Result | RL | MDL | Units | Q |
|----------|-------------|-----------------|------|-------|-------|---|
| 91-20-3 | Naphthalene | ND ^a | 0.10 | 0.030 | ug/l | |
| 123-91-1 | 1,4-Dioxane | 55.5 | 1.0 | 0.050 | ug/l | |

| CAS No. | Surrogate Recoveries | Run# 1 | Run# 2 | Limits |
|-----------|----------------------|--------|--------|---------|
| 4165-60-0 | Nitrobenzene-d5 | 69% | 60% | 24-125% |
| 321-60-8 | 2-Fluorobiphenyl | 66% | 51% | 19-127% |
| 1718-51-0 | Terphenyl-d14 | 70% | 82% | 10-119% |

(a) Result is from Run# 2



ND = Not detected MDL = Method Detection Limit
 RL = Reporting Limit
 E = Indicates value exceeds calibration range

J = Indicates an estimated value
 B = Indicates analyte found in associated method blank
 N = Indicates presumptive evidence of a compound

Matrix Spike/Matrix Spike Duplicate Summary

Page 1 of 1

Job Number: JC25442

Account: AMANYWP Anderson, Mulholland & Associates

Project: BSMC, Building 5 Area, PR

| Sample | File ID | DF | Analyzed | By | Prep Date | Prep Batch | Analytical Batch |
|--------------|-----------|----|----------|----|-----------|------------|------------------|
| OP96175A-MS | 4M67274.D | 1 | 08/10/16 | JJ | 08/09/16 | OP96175A | E4M3041 |
| OP96175A-MSD | 4M67275.D | 1 | 08/10/16 | JJ | 08/09/16 | OP96175A | E4M3041 |
| JC25442-2 | F159724.D | 1 | 08/10/16 | AD | 08/09/16 | OP96175A | EF6717 |
| JC25442-2 | 4M67273.D | 1 | 08/10/16 | JJ | 08/09/16 | OP96175A | E4M3041 |

The QC reported here applies to the following samples:

Method: SW846 8270D BY SIM

JC25442-1, JC25442-2, JC25442-3, JC25442-4

| CAS No. | Compound | JC25442-2 ug/l | Spike Q ug/l | MS ug/l | MS % | Spike ug/l | MSD ug/l | MSD % | RPD | Limits Rec/RPD |
|----------|-------------|-------------------|-----------------|------------|-----------------|---------------|-------------|-----------------|-----|-------------------|
| 91-20-3 | Naphthalene | ND ^a | 2.04 | 1.54 | 75 | 2.04 | 1.59 | 78 | 3 | 23-140/36 |
| 123-91-1 | 1,4-Dioxane | 49.8 | 2.04 | 44.8 | 0* ^b | 2.04 | 42.7 | 0* ^b | 5 | 20-160/30 |

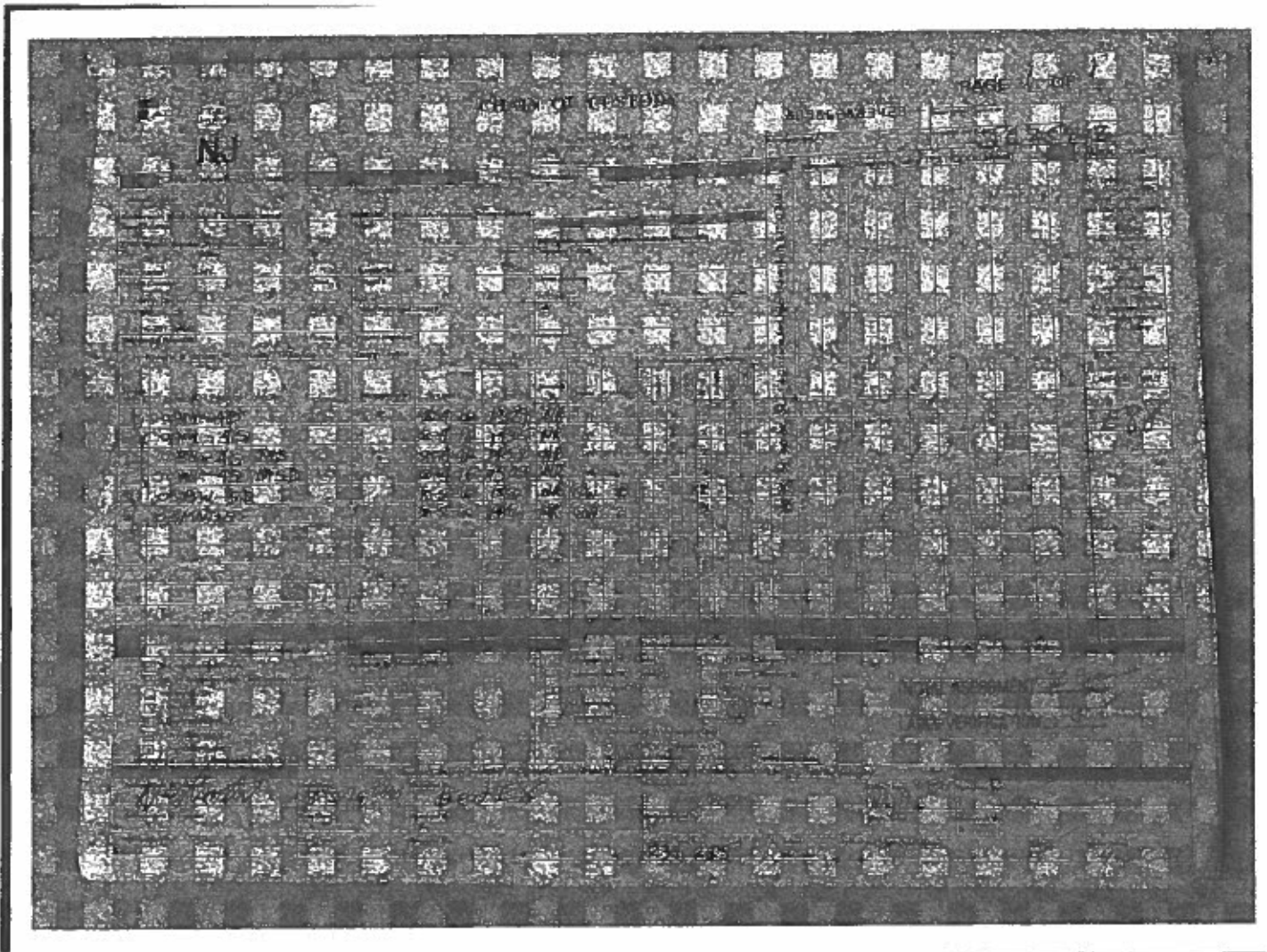
| CAS No. | Surrogate Recoveries | MS | MSD | JC25442-2 | JC25442-2 | Limits |
|-----------|----------------------|------|------|-----------|-----------|---------|
| 367-12-4 | 2-Fluorophenol | 64% | 63% | | | 14-81% |
| 4165-62-2 | Phenol-d5 | 54% | 53% | | | 11-54% |
| 118-79-6 | 2,4,6-Tribromophenol | 115% | 118% | | | 35-145% |
| 4165-60-0 | Nitrobenzene-d5 | 75% | 75% | 73% | 65% | 24-125% |
| 321-60-8 | 2-Fluorobiphenyl | 61% | 63% | 69% | 55% | 19-127% |
| 1718-51-0 | Terphenyl-d14 | 102% | 111% | 76% | 91% | 10-119% |

(a) Result is from Run #2.

(b) Outside control limits due to high level in sample relative to spike amount.



* = Outside of Control Limits.



JC25442: Chain of Custody
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EXECUTIVE NARRATIVE

SDG No: JC25442 Laboratory: Accutest, New Jersey
Analysis: SW846-8270D Number of Samples: 6
Location: BMSMC, Building 5 Area, PR
Humacao, PR

SUMMARY: Six (6) samples were analyzed for Naphthalene and 1,4-Dioxane following method SW846-8270D using the selective ion monitoring (SIM) technique; four of the samples were also analyzed for 1,4-Dioxane following method SW846-8270D in the scan mode. The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: EPA Hazardous Waste Support Section, SOP HW-35A, July 2015 –Revision 0. Semivolatile Data Validation. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

Results are valid and can be used for decision making purposes.

Critical issues: None
Major: None
Minor: None

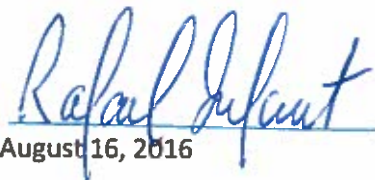
Critical findings: None
Major findings: None
Minor findings: 1. MS/MSD % recoveries and RPD within laboratory control limits except in the cases described in the Data Review Worksheet. No action taken, MS/MSD % recovery outside control limits due to high level in sample relative to spike amount.

COMMENTS: Results are valid and can be used for decision making purposes.

Reviewers Name: Rafael Infante
Chemist License 1888

Signature:

Date:


August 16, 2016

SAMPLE ORGANIC DATA SAMPLE SUMMARY

Sample ID: JC25442-1
Sample location: BMSMC, Building 5 Area, PR
Sampling date: 8/5/2016
Matrix: Groundwater

METHOD: 8270D (SIM)

| Analyte Name | Result | Units | Dilution Factor | Lab Flag | Validation | Reportable |
|--------------|--------|-------|-----------------|----------|------------|------------|
| Naphthalene | 0.10 | ug/l | 1 | - | U | Yes |
| 1,4-Dioxane | - | - | - | - | - | - |

METHOD: 8270D (SCAN)

| Analyte Name | Result | Units | Dilution Factor | Lab Flag | Validation | Reportable |
|--------------|--------|-------|-----------------|----------|------------|------------|
| 1,4-Dioxane | 28.4 | ug/l | 1 | - | - | Yes |

Sample ID: JC25442-2
Sample location: BMSMC, Building 5 Area, PR
Sampling date: 8/5/2016
Matrix: Groundwater

METHOD: 8270D (SIM)

| Analyte Name | Result | Units | Dilution Factor | Lab Flag | Validation | Reportable |
|--------------|--------|-------|-----------------|----------|------------|------------|
| Naphthalene | 0.10 | ug/l | 1 | - | U | Yes |
| 1,4-Dioxane | - | - | - | - | - | - |

METHOD: 8270D (SCAN)

| Analyte Name | Result | Units | Dilution Factor | Lab Flag | Validation | Reportable |
|--------------|--------|-------|-----------------|----------|------------|------------|
| 1,4-Dioxane | 49.8 | ug/l | 1 | - | - | Yes |

Sample ID: JC25442-3
Sample location: BMSMC, Building 5 Area, PR
Sampling date: 8/5/2016
Matrix: Groundwater

METHOD: 8270D (SIM)

| Analyte Name | Result | Units | Dilution Factor | Lab Flag | Validation | Reportable |
|--------------|--------|-------|-----------------|----------|------------|------------|
| Naphthalene | 0.10 | ug/l | 1 | - | U | Yes |
| 1,4-Dioxane | - | - | - | - | - | - |

Sample ID: JC25442-3
Sample location: BMSMC, Building 5 Area, PR
Sampling date: 8/5/2016
Matrix: Groundwater

METHOD: 8270D (SCAN)

| Analyte Name | Result | Units | Dilution Factor | Lab Flag | Validation | Reportable |
|--------------|--------|-------|-----------------|----------|------------|------------|
| 1,4-Dioxane | 55.2 | ug/l | 1 | - | - | Yes |

Sample ID: JC25442-4
Sample location: BMSMC, Building 5 Area, PR
Sampling date: 8/5/2016
Matrix: Groundwater

METHOD: 8270D (SIM)

| Analyte Name | Result | Units | Dilution Factor | Lab Flag | Validation | Reportable |
|--------------|--------|-------|-----------------|----------|------------|------------|
| Naphthalene | 0.10 | ug/l | 1 | - | U | Yes |
| 1,4-Dioxane | - | - | - | - | - | - |

METHOD: 8270D (SCAN)

| Analyte Name | Result | Units | Dilution Factor | Lab Flag | Validation | Reportable |
|--------------|--------|-------|-----------------|----------|------------|------------|
| 1,4-Dioxane | 55.5 | ug/l | 1 | - | - | Yes |

Sample ID: JC25442-2MS
Sample location: BMSMC, Building 5 Area, PR
Sampling date: 8/5/2016
Matrix: Groundwater

METHOD: 8270D (SIM)

| Analyte Name | Result | Units | Dilution Factor | Lab Flag | Validation | Reportable |
|--------------|--------|-------|-----------------|----------|------------|------------|
| Naphthalene | 1.54 | ug/l | 1 | - | - | Yes |
| 1,4-Dioxane | 44.8 | ug/l | 1 | - | - | Yes |

Sample ID: JC25442-2MSD
Sample location: BMSMC, Building 5 Area, PR
Sampling date: 8/5/2016
Matrix: Groundwater

METHOD: 8270D (SIM)

| Analyte Name | Result | Units | Dilution Factor | Lab Flag | Validation | Reportable |
|--------------|--------|-------|-----------------|----------|------------|------------|
| Naphthalene | 1.59 | ug/l | 1 | - | - | Yes |
| 1,4-Dioxane | 42.7 | ug/l | 1 | - | - | Yes |

DATA REVIEW WORKSHEETS

Project Number: JC25442
 Date: August 5, 2016
 Shipping Date: August 5, 2016
 EPA Region: 2

REVIEW OF SEMIVOLATILE ORGANIC PACKAGE

The following guidelines for evaluating volatile organics were created to delineate required validation actions. This document will assist the reviewer in using professional judgment to make more informed decision and in better serving the needs of the data users. The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: EPA Hazardous Waste Support Section, SOP HW-35A, July 2015 –Revision 0. *Semivolatile Data Validation*. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

The hardcopied (laboratory name) Accutest data package received has been reviewed and the quality control and performance data summarized. The data review for SVOCs included:

Lab. Project/SDG No.: JC25442 Sample matrix: Groundwater
 No. of Samples: 6 SIM/4 SCAN
 Trip blank No.: -
 Field blank No.: -
 Equipment blank No.: -
 Field duplicate No.: -

| | |
|---|---|
| <input checked="" type="checkbox"/> Data Completeness | <input checked="" type="checkbox"/> Laboratory Control Spikes |
| <input checked="" type="checkbox"/> Holding Times | <input checked="" type="checkbox"/> Field Duplicates |
| <input checked="" type="checkbox"/> GC/MS Tuning | <input checked="" type="checkbox"/> Calibrations |
| <input checked="" type="checkbox"/> Internal Standard Performance | <input checked="" type="checkbox"/> Compound Identifications |
| <input checked="" type="checkbox"/> Blanks | <input checked="" type="checkbox"/> Compound Quantitation |
| <input checked="" type="checkbox"/> Surrogate Recoveries | <input checked="" type="checkbox"/> Quantitation Limits |
| <input checked="" type="checkbox"/> Matrix Spike/Matrix Spike Duplicate | |

Overall Comments: Naphthalene and 1,4-Dioxane analyzed by method SW846-8270D (SIM);
Sample JC25442-1 to JC25442-4 also analyzed by the scan method

Definition of Qualifiers:

J- Estimated results
 U- Compound not detected
 R- Rejected data
 UJ- Estimated nondetect

Reviewer: Rafael Infante
 Date: August 16, 2016

DATA REVIEW WORKSHEETS

DATA COMPLETENESS

MISSING INFORMATION

DATE LAB. CONTACTED

DATE RECEIVED

[illegible]

DATA REVIEW WORKSHEETS

All criteria were met X
 Criteria were not met
 and/or see below

HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

| SAMPLE ID | DATE SAMPLED | DATE EXTRACTED/ANALYZED | pH | ACTION |
|---|--------------|-------------------------|----|--------|
| | | | | |
| All samples extracted and analyzed within method recommended holding time. Samples properly preserved except in the cases described in this document. | | | | |
| | | | | |

Cooler temperature (Criteria: 4 ± 2 °C): 5.4°C

Actions

Results will be qualified based on the criteria of the following Table:

Table 1. Holding Time Actions for Semivolatile Analyses

| Matrix | Preserved | Criteria | Action | |
|-------------|-----------|--|-------------------------------|-----------------------------------|
| | | | Detected Associated Compounds | Non-Detected Associated Compounds |
| Aqueous | No | ≤ 7 days (for extraction) ≤ 40 days (for analysis) | Use professional judgment | |
| | No | > 7 days (for extraction) > 40 days (for analysis) | J | Use professional judgment |
| | Yes | ≤ 7 days (for extraction) ≤ 40 days (for analysis) | No qualification | |
| | Yes | > 7 days (for extraction) > 40 days (for analysis) | J | UJ |
| | Yes/No | Grossly Exceeded | J | UJ or R |
| Non-Aqueous | No | ≤ 14 days (for extraction) ≤ 40 days (for analysis) | Use professional judgment | |
| | No | > 14 days (for extraction) > 40 days (for analysis) | J | Use professional judgment |
| | Yes | ≤ 14 days (for extraction) ≤ 40 days (for analysis) | No qualification | |
| | Yes | > 14 days (for extraction) > 40 days (for analysis) | J | UJ |
| | Yes/No | Grossly Exceeded | J | UJ or R |

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met see below

GC/MS TUNING

The assessment of the tuning results is to determine if the sample instrumentation is within the standard tuning QC limits

 X The DFTPP performance results were reviewed and found to be within the specified criteria.

 X DFTPP tuning was performed for every 12 hours of sample analysis.

If no, use professional judgment to determine whether the associated data should be accepted, qualified or rejected.

Notes: These requirements do not apply when samples are analyzed by the Selected Ion Monitoring (SIM) technique.

All mass spectrometer conditions must be identical to those used during the sample analysis. Background subtraction actions resulting in spectral distortion are unacceptable

Notes: No data should be qualified based of DFTPP failure.

The requirement to analyze the instrument performance check solution is optional when analysis of PAHs/pentachlorophenol is to be performed by the SIM technique.

List the samples affected:

Actions:

1. If sample are analyzed without a preceding valid instrument performance check or are analyzed 12 hours after the Instrument Performance Check, qualify all data in those samples as unusable (R).
2. If ion abundance criteria are not met, use professional judgment to determine to what extent the data may be utilized.
3. State in the Data Review Narrative, decisions to use analytical data associated with DFTPP instrument performance checks not meeting the contract requirements.
4. Use professional judgment to determine if associated data should be qualified based on the spectrum of the mass calibration compounds.

DATA REVIEW WORKSHEETS

All criteria were met X
 Criteria were not met _____
 and/or see below _____

INITIAL CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration: 08/03/16 (SIM)
 Instrument ID numbers: GCMS4M
 Matrix/Level: Aqueous/low

Date of initial calibration: 08/03/16 (SCAN)
 Instrument ID numbers: GCMSF
 Matrix/Level: Aqueous/low

| DATE | LAB ID# | FILE | CRITERIA OUT RFs, %RSD, %D, r | COMPOUND | SAMPLES AFFECTED |
|--|---------|------|----------------------------------|----------|---------------------|
| | | | | | |
| Initial and initial calibration verification meets the method and guidance validation document performance criteria. | | | | | |
| | | | | | |

Note:

Actions:

Qualify the initial calibration analytes listed in Table 2 using the following criteria:

Table 3. Initial Calibration Actions for Semivolatile Analysis

| Criteria | Action | |
|---|--------------------------------------|--------------------------------|
| | Detect | Non-detect |
| Initial Calibration not performed at specified frequency and sequence | Use professional judgment R | Use professional judgment R |
| Initial Calibration not performed at the specified concentrations | J | UJ |
| RRF < Minimum RRF in Table 2 for target analyte | Use professional judgment J+ or R | R |
| RRF ≥ Minimum RRF in Table 2 for target analyte | No qualification | No qualification |
| %RSD > Maximum %RSD in Table 2 for target analyte | J | Use professional judgment |
| %RSD ≤ Maximum %RSD in Table 2 for target analyte | No qualification | No qualification |

DATA REVIEW WORKSHEETS

Initial Calibration

Table 2. RRF, %RSD, and %D Acceptance Criteria in Initial Calibration and CCV for Semivolatile Analysis

| Analyte | Minimum RRF | Maximum %RSD | Opening Maximum %D ¹ | Opening Maximum %D ¹ |
|-------------------------------|-------------|--------------|---------------------------------|---------------------------------|
| 1,4-Dioxane | 0.010 | 40.0 | ± 40.0 | ± 50.0 |
| Benzaldehyde | 0.100 | 40.0 | ± 40.0 | ± 50.0 |
| Phenol | 0.080 | 20.0 | ± 20.0 | ± 25.0 |
| Bis(2-chloroethyl)ether | 0.100 | 20.0 | ± 20.0 | ± 25.0 |
| 2-Chlorophenol | 0.200 | 20.0 | ± 20.0 | ± 25.0 |
| 2-Methylphenol | 0.010 | 20.0 | ± 20.0 | ± 25.0 |
| 3-Methylphenol | 0.010 | 20.0 | ± 20.0 | ± 25.0 |
| 2,2'-Oxybis-(1-chloropropane) | 0.010 | 20.0 | ± 25.0 | ± 50.0 |
| Acetophenone | 0.060 | 20.0 | ± 20.0 | ± 25.0 |
| 4-Methylphenol | 0.010 | 20.0 | ± 20.0 | ± 25.0 |
| N-Nitroso-di-n-propylamine | 0.080 | 20.0 | ± 25.0 | ± 25.0 |
| Hexachloroethane | 0.100 | 20.0 | ± 20.0 | ± 25.0 |
| Nitrobenzene | 0.090 | 20.0 | ± 20.0 | ± 25.0 |
| Isophorone | 0.100 | 20.0 | ± 20.0 | ± 25.0 |
| 2-Nitrophenol | 0.060 | 20.0 | ± 20.0 | ± 25.0 |
| 2,4-Dimethylphenol | 0.050 | 20.0 | ± 25.0 | ± 50.0 |
| Bis(2-chloroethoxy)methane | 0.080 | 20.0 | ± 20.0 | ± 25.0 |
| 2,4-Dichlorophenol | 0.060 | 20.0 | ± 20.0 | ± 25.0 |
| Naphthalene | 0.200 | 20.0 | ± 20.0 | ± 25.0 |
| 4-Chloroaniline | 0.010 | 40.0 | ± 40.0 | ± 50.0 |
| Hexachlorobutadiene | 0.040 | 20.0 | ± 20.0 | ± 25.0 |
| Caprolactam | 0.010 | 40.0 | ± 30.0 | ± 50.0 |
| 4-Chloro-3-methylphenol | 0.040 | 20.0 | ± 20.0 | ± 25.0 |
| 2-Methylnaphthalene | 0.100 | 20.0 | ± 20.0 | ± 25.0 |
| Hexachlorocyclopentadiene | 0.010 | 40.0 | ± 40.0 | ± 50.0 |
| 2,4,6-Trichlorophenol | 0.090 | 20.0 | ± 20.0 | ± 25.0 |
| 2,4,5-Trichlorophenol | 0.100 | 20.0 | ± 20.0 | ± 25.0 |
| 1,1'-Biphenyl | 0.200 | 20.0 | ± 20.0 | ± 25.0 |

DATA REVIEW WORKSHEETS

| Analyte | Minimum RRF | Maximum %RSD | Opening Maximum %D ¹ | Opening Maximum %D ¹ |
|----------------------------|-------------|--------------|---------------------------------|---------------------------------|
| 2-Chloronaphthalene | 0.300 | 20.0 | ± 20.0 | ± 25.0 |
| 2-Nitroaniline | 0.060 | 20.0 | ± 25.0 | ± 25.0 |
| Dimethylphthalate | 0.300 | 20.0 | ± 25.0 | ± 25.0 |
| 2,6-Dinitrotoluene | 0.080 | 20.0 | ± 20.0 | ± 25.0 |
| Acenaphthylene | 0.400 | 20.0 | ± 20.0 | ± 25.0 |
| 3-Nitroaniline | 0.010 | 20.0 | ± 25.0 | ± 50.0 |
| Acenaphthene | 0.200 | 20.0 | ± 20.0 | ± 25.0 |
| 2,4-Dinitrophenol | 0.010 | 40.0 | ± 50.0 | ± 50.0 |
| 4-Nitrophenol | 0.010 | 40.0 | ± 40.0 | ± 50.0 |
| Dibenzofuran | 0.300 | 20.0 | ± 20.0 | ± 25.0 |
| 2,4-Dinitrotoluene | 0.070 | 20.0 | ± 20.0 | ± 25.0 |
| Diethylphthalate | 0.300 | 20.0 | ± 20.0 | ± 25.0 |
| 1,2,4,5-Tetrachlorobenzene | 0.100 | 20.0 | ± 20.0 | ± 25.0 |
| 4-Chlorophenyl-phenylether | 0.100 | 20.0 | ± 20.0 | ± 25.0 |
| Fluorene | 0.200 | 20.0 | ± 20.0 | ± 25.0 |
| 4-Nitroaniline | 0.010 | 40.0 | ± 40.0 | ± 50.0 |
| 4,6-Dinitro-2-methylphenol | 0.010 | 40.0 | ± 30.0 | ± 50.0 |
| 4-Bromophenyl-phenyl ether | 0.070 | 20.0 | ± 20.0 | ± 25.0 |
| N-Nitrosodiphenylamine | 0.100 | 20.0 | ± 20.0 | ± 25.0 |
| Hexachlorobenzene | 0.050 | 20.0 | ± 20.0 | ± 25.0 |
| Atrazine | 0.010 | 40.0 | ± 25.0 | ± 50.0 |
| Pentachlorophenol | 0.010 | 40.0 | ± 40.0 | ± 50.0 |
| Phenanthrene | 0.200 | 20.0 | ± 20.0 | ± 25.0 |
| Anthracene | 0.200 | 20.0 | ± 20.0 | ± 25.0 |
| Carbazole | 0.050 | 20.0 | ± 20.0 | ± 25.0 |
| Di-n-butylphthalate | 0.500 | 20.0 | ± 20.0 | ± 25.0 |
| Fluoranthene | 0.100 | 20.0 | ± 20.0 | ± 25.0 |
| Pyrene | 0.400 | 20.0 | ± 25.0 | ± 50.0 |
| Butylbenzylphthalate | 0.100 | 20.0 | ± 25.0 | ± 50.0 |

DATA REVIEW WORKSHEETS

| Analyte | Minimum RRF | Maximum %RSD | Opening Maximum %D' | Opening Maximum %D' |
|-----------------------------|----------------|-----------------|---------------------------|---------------------------|
| 3,3'-Dichlorobenzidine | 0.010 | 40.0 | ± 40.0 | ± 50.0 |
| Benzo(a)anthracene | 0.300 | 20.0 | ± 20.0 | ± 25.0 |
| Chrysene | 0.200 | 20.0 | ± 20.0 | ± 50.0 |
| Bis(2-ethylhexyl) phthalate | 0.200 | 20.0 | ± 25.0 | ± 50.0 |
| Di-n-octylphthalate | 0.010 | 40.0 | ± 40.0 | ± 50.0 |
| Benzo(b)fluoranthene | 0.010 | 20.0 | ± 25.0 | ± 50.0 |
| Benzo(k)fluoranthene | 0.010 | 20.0 | ± 25.0 | ± 50.0 |
| Benzo(a)pyrene | 0.010 | 20.0 | ± 20.0 | ± 50.0 |
| Indeno(1,2,3-cd)pyrene | 0.010 | 20.0 | ± 25.0 | ± 50.0 |
| Dibenzo(a,h)anthracene | 0.010 | 20.0 | ± 25.0 | ± 50.0 |
| Benzo(g,h,i)perylene | 0.010 | 20.0 | ± 30.0 | ± 50.0 |
| 2,3,4,6-Tetrachlorophenol | 0.040 | 20.0 | ± 20.0 | ± 50.0 |
| Naphthalene | 0.600 | 20.0 | ± 25.0 | ± 25.0 |
| 2-Methylnaphthalene | 0.300 | 20.0 | ± 20.0 | ± 25.0 |
| Acenaphthylene | 0.900 | 20.0 | ± 20.0 | ± 25.0 |
| Acenaphthene | 0.500 | 20.0 | ± 20.0 | ± 25.0 |
| Fluorene | 0.700 | 20.0 | ± 25.0 | ± 50.0 |
| Phenanthrene | 0.300 | 20.0 | ± 25.0 | ± 50.0 |
| Anthracene | 0.400 | 20.0 | ± 25.0 | ± 50.0 |
| Fluoranthene | 0.400 | 20.0 | ± 25.0 | ± 50.0 |
| Pyrene | 0.500 | 20.0 | ± 30.0 | ± 50.0 |
| Benzo(a)anthracene | 0.400 | 20.0 | ± 25.0 | ± 50.0 |
| Chrysene | 0.400 | 20.0 | ± 25.0 | ± 50.0 |
| Benzo(b)fluoranthene | 0.100 | 20.0 | ± 30.0 | ± 50.0 |
| Benzo(k)fluoranthene | 0.100 | 20.0 | ± 30.0 | ± 50.0 |
| Benzo(a)pyrene | 0.100 | 20.0 | ± 25.0 | ± 50.0 |
| Indeno(1,2,3-cd)pyrene | 0.100 | 20.0 | ± 40.0 | ± 50.0 |
| Dibenzo(a,h)anthracene | 0.010 | 25.0 | ± 40.0 | ± 50.0 |
| Benzo(g,h,i)perylene | 0.020 | 25.0 | ± 40.0 | ± 50.0 |

DATA REVIEW WORKSHEETS

| Pentachlorophenol | 0.010 | 40.0 | ± 50.0 | ± 50.0 |
|---|-------------|--------------|---------------------------------|--------------------|
| Deuterated Monitoring Compounds | | | | |
| Analyte | Minimum RRF | Maximum %RSD | Opening Maximum %D ¹ | Closing Maximum %D |
| 1,4-Dioxane-d ₈ | 0.010 | 20.0 | ± 25.0 | ± 50.0 |
| Phenol-d ₅ | 0.010 | 20.0 | ± 25.0 | ± 25.0 |
| Bis-(2-chloroethyl)ether-d ₈ | 0.100 | 20.0 | ± 20.0 | ± 25.0 |
| 2-Chlorophenol-d ₄ | 0.200 | 20.0 | ± 20.0 | ± 25.0 |
| 4-Methylphenol-d ₈ | 0.010 | 20.0 | ± 20.0 | ± 25.0 |
| 4-Chloroaniline-d ₄ | 0.010 | 40.0 | ± 40.0 | ± 50.0 |
| Nitrobenzene-d ₅ | 0.050 | 20.0 | ± 20.0 | ± 25.0 |
| 2-Nitrophenol-d ₄ | 0.050 | 20.0 | ± 20.0 | ± 25.0 |
| 2,4-Dichlorophenol-d ₃ | 0.060 | 20.0 | ± 20.0 | ± 25.0 |
| Dimethylphthalate-d ₆ | 0.300 | 20.0 | ± 20.0 | ± 25.0 |
| Acenaphthylene-d ₈ | 0.400 | 20.0 | ± 20.0 | ± 25.0 |
| 4-Nitrophenol-d ₄ | 0.010 | 40.0 | ± 40.0 | ± 50.0 |
| Fluorene-d ₁₀ | 0.100 | 20.0 | ± 20.0 | ± 25.0 |
| 4,6-Dinitro-2-methylphenol-d ₂ | 0.010 | 40.0 | ± 30.0 | ± 50.0 |
| Anthracene-d ₁₀ | 0.300 | 20.0 | ± 20.0 | ± 25.0 |
| Pyrene-d ₁₀ | 0.300 | 20.0 | ± 25.0 | ± 50.0 |
| Benzo(a)pyrene-d ₁₂ | 0.010 | 20.0 | ± 20.0 | ± 50.0 |
| Fluoranthene-d ₁₀ (SIM) | 0.400 | 20.0 | ± 25.0 | ± 50.0 |
| 2-Methylnaphthalene-d ₁₀ (SIM) | 0.300 | 20.0 | ± 20.0 | ± 25.0 |

¹ If a closing CCV is acting as an opening CCV, all target analytes must meet the requirements for an opening CCV.

Note: If analysis by SIM technique is requested for PAH/pentachlorophenols, calibration standards analyzed at 0.10, 0.20, 0.40, 0.80, and 1.0 ng/uL for each target compound of interest and the associated DMCs. Pentachlorophenol will require only a four point initial calibration at 0.20, 0.40, 0.80, and 1.0 ng/uL.

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met
and/or see below _____

CONTINUING CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration: 08/03/16 (SIM)
Date of initial calibration verification (ICV): 08/03-04/16
Date of continuing calibration verification (CCV): 08/10/16
Date of closing CCV: -
Instrument ID numbers: GCMS4M
Matrix/Level: Aqueous/low

Date of initial calibration: 08/03/16 (Scan)
Date of initial calibration verification (ICV): 08/03/16
Date of continuing calibration verification (CCV): 08/10/16
Date of closing CCV: -
Instrument ID numbers: GCMSF
Matrix/Level: Aqueous/low

| DATE | LAB FILE ID# | CRITERIA OUT RFs, %RSD, %D, r | COMPOUND | SAMPLES AFFECTED |
|------|--------------|-------------------------------|----------|------------------|
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |

Note: Initial and continuing calibration verifications meet the method and guidance document required performance criteria. No closing calibration verification included in data package. No action taken, professional judgment.

Actions:

Notes: Verify that the CCV is run at the required frequency (an opening and closing CCV must be run within 12-hour period).

All DMCs must meet the RRF values given in Table 2. No qualification of the data is necessary on DMCs RRF and %RSD/%D alone. Use professional judgment to evaluate DMCs and %RSD/%D data in conjunction with DMCs recoveries to determine the need for qualification of the data.

Qualify the initial calibration analytes listed in Table 2 using the following criteria in the CCVs:

DATA REVIEW WORKSHEETS

Table 4. CCV Actions for Semivolatile Analysis

| Criteria for Opening CCV | Criteria for Closing CCV | Action | |
|---|---|-------------------------------------|--------------------------------|
| | | Detect | Non-detect |
| CCV not performed at required frequency and sequence | CCV not performed at required frequency | Use professional judgment R | Use professional judgment R |
| CCV not performed at specified concentration | CCV not performed at specified concentration | Use professional judgment | Use professional judgment |
| RRF < Minimum RRF in Table 2 for target analyte | RRF < Minimum RRF in Table 2 for target analyte | Use professional judgment J or R | R |
| RRF \geq Minimum RRF in Table 2 for target analyte | RRF \geq Minimum RRF in Table 2 for target analyte | No qualification | No qualification |
| %D outside the Opening Maximum %D limits in Table 2 for target analyte | %D outside the Closing Maximum %D limits in Table 2 for target analyte | J | UJ |
| %D within the inclusive Opening Maximum %D limits in Table 2 for target analyte | %D within the inclusive Closing Maximum %D limits in Table 2 for target analyte | No qualification | No qualification |

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met
and/or see below _____

BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

List the contamination in the blanks below. High and low levels blanks must be treated separately.

Notes: The concentration of non-target compounds in all blanks must be less than or equal to 10 ug/L.

The concentration of target compounds in all blanks must be less than its CRQL listed in the method.

Samples taken from a drinking water tap do not have an associated field blank.

Laboratory blanks

| DATE ANALYZED | LAB ID | LEVEL/ MATRIX | COMPOUND | CONCENTRATION UNITS |
|---|--------|------------------|----------|------------------------|
| _No_target_analytes_detected_in_method_blanks._ | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |

Field/Equipment/Trip blank

| DATE ANALYZED | LAB ID | LEVEL/ MATRIX | COMPOUND | CONCENTRATION UNITS |
|---|--------|------------------|----------|------------------------|
| _No_field/trip/equipment_blanks_analyzed_with_this_data_package._ | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |
| | | | | |

Note:

DATA REVIEW WORKSHEETS

All criteria were met X
 Criteria were not met
 and/or see below

BLANK ANALYSIS RESULTS (Section 3)

Blank Actions

Qualify samples based on the criteria summarized in Table 5:

Table 5. Blank and TCLP/SPLP LEB Actions for Semivolatile Analysis

| Blank Type | Blank Result | Sample Result | Action |
|------------------------------------|---|---------------------------|---|
| Method, TCLP/SPLP LEB, Field | Detect | Non-detect | No qualification |
| | < CRQL | < CRQL | Report at CRQL and qualify as non-detect (U) |
| | | ≥ CRQL | Use professional judgment |
| | ≥ CRQL | < CRQL | Report at CRQL and qualify as non-detect (U) |
| | | ≥ CRQL but < Blank Result | Report at sample results and qualify as non-detect (U) or as unusable (R) |
| | | ≥ CRQL and ≥ Blank Result | Use professional judgment |
| | Grossly high | Detect | Report at sample results and qualify as unusable (R) |
| | TIC > 5.0 ug/L (water) or 0.0050 mg/L (TCLP leachate) or TIC > 170 ug/Kg (soil) | Detect | Use professional judgment |

List samples qualified

| CONTAMINATION SOURCE/LEVEL | COMPOUND | CONC/UNITS | AL/UNITS | SQL | AFFECTED SAMPLES |
|----------------------------|----------|------------|----------|-----|------------------|
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |
| | | | | | |

DATA REVIEW WORKSHEETS

All criteria were met X
 Criteria were not met
 and/or see below

SURROGATE SPIKE RECOVERIES – DEUTERATED MONITORING COMPOUNDS (DMCs)

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries – deuterated monitoring compounds. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

Notes: Recoveries for DMCs in samples and blanks must be within the limits specified in Table 6.

The recovery limits for any of the compounds listed in Table 6 may be expanded at any time during the period of performance if USEPA determines that the limits are too restrictive.

If a DMC is not added in the samples and blanks or the concentrations of DMCs in the samples and blank not the specified, use professional judgment in qualifying the data.

Table 7. DMC Actions for Semivolatile Analysis

| Criteria | Action | |
|---|------------------|------------------|
| | Detect | Non-detect |
| %R < 10% (excluding DMCs with 10% as a lower acceptance limit) | J- | R |
| 10% ≤ %R (excluding DMCs with 10% as a lower acceptance limit) < Lower Acceptance Limit | J- | UJ |
| Lower Acceptance limit ≤ %R ≤ Upper Acceptance Limit | No qualification | No qualification |
| %R > Upper Acceptance Limit | J+ | No qualification |

List the percent recoveries (%Rs) which do not meet the criteria for DMCs (surrogate) recovery.

Matrix: Groundwater

SAMPLE ID

SURROGATE COMPOUND

ACTION

DMCs meet the required criteria. Non-deuterated surrogates added to the samples were
within laboratory recovery limits.

DATA REVIEW WORKSHEETS

Table 8. Semivolatile DMCs and the Associated Target Analytes

| 1,4-Dioxane-d₈ (DMC-1) | Phenol-d₅ (DMC-2) | Bis(2-Chloroethyl) ether-d₈ (DMC-3) |
|--|---|--|
| 1,4-Dioxane | Benzaldehyde Phenol | Bis(2-chloroethyl) ether 2,2'-Oxybis(1-chloropropane) Bis(2-chloroethoxy) methane |
| 2-Chlorophenol-d₄ (DMC-4) | 4-Methylphenol-d₄ (DMC-5) | 4-Chloroaniline-d₄ (DMC-6) |
| 2-Chlorophenol | 2-Methylphenol 3-Methylphenol 4-Methylphenol 2,4-Dimethylphenol | 4-Chloroaniline Hexachlorocyclopentadiene Dichlorobenzidine |
| Nitrobenzene-d₅ (DMC-7) | 2-Nitrophenol-d₄ (DMC-8) | 2,4-Dichlorophenol-d₃ (DMC-9) |
| Acetophenone N-Nitroso-di-n-propylamine Hexachloroethane Nitrobenzene 2,6-Dinitrotoluene 2,4-Dinitrotoluene N-Nitrosodiphenylamine | Isophorone 2-Nitrophenol | 2,4-Dichlorophenol Hexachlorobutadiene Hexachlorocyclopentadiene 4-Chloro-3-methylphenol 2,4,6-Trichlorophenol 2,4,5-Trichlorophenol 1,2,4,5-Tetrachlorobenzene *Pentachlorophenol 2,3,4,6-Tetrachlorophenol |
| Dimethylphthalate-d₆ (DMC-10) | Acenaphthylene-d₈ (DMC-11) | 4-Nitrophenol-d₄ (DMC-12) |
| Caprolactam 1,1'-Biphenyl Dimethylphthalate Diethylphthalate Di-n-butylphthalate Butylbenzylphthalate Bis(2-ethylhexyl) phthalate Di-n-octylphthalate | *Naphthalene *2-Methylnaphthalene 2-Chloronaphthalene *Acenaphthylene *Acenaphthene | 2-Nitroaniline 3-Nitroaniline 2,4-Dinitrophenol 4-Nitrophenol 4-Nitroaniline |

DATA REVIEW WORKSHEETS

| | | |
|---|--|---|
| Fluorene-d₁₀ (DMC-13) | 4,6-Dinitro-2-methylphenol-d₂ (DMC-14) | Anthracene-d₁₀ (DMC-15) |
| Dibenzofuran *Fluorene 4-Chlorophenyl-phenylether 4-Bromophenyl-phenylether Carbazole | 4,6-Dinitro-2-methylphenol | Hexachlorobenzene Atrazine *Phenanthrene *Anthracene |
| Pyrene-d₁₀ (DMC-16) | Benzo(a)pyrene-d₁₂ (DMC-17) | |
| *Fluoranthene *Pyrene *Benzo(a)anthracene *Chrysene | 3,3'-Dichlorobenzidine *Benzo(b)fluoranthene *Benzo(k)fluoranthene *Benzo(a)pyrene *Indeno(1,2,3-cd)pyrene *Dibenzo(a,h)anthracene *Benzo(g,h,i)perylene | |

*Included in optional Target Analyte List (TAL) of PAHs and PCP only.

Table 9. Semivolatile SIM DMCs and the Associated Target Analytes

| Fluoranthene-d₁₀ (DMC-1) | 2-Methylnaphthalene-d₁₀ (DMC-2) |
|--|---|
| Fluoranthene | Naphthalene |
| Pyrene | 2-Methylnaphthalene |
| Benzo(a)anthracene | Acenaphthylene |
| Chrysene | Acenaphthene |
| Benzo(b)fluoranthene | Fluorene |
| Benzo(k)fluoranthene | Pentachlorophenol |
| Benzo(a)pyrene | Phenanthrene |
| Indeno(1,2,3-cd)pyrene | Anthracene |
| Dibenzo(a,h)anthracene | |
| Benzo(g,h,i)perylene | |

DATA REVIEW WORKSHEETS

All criteria were met _____
 Criteria were not met _____
 and/or see below _____X_____

VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples. If any % R in the MS or MSD falls outside the designated range, the reviewer should determine if there are matrix effects, i.e. LCS data are within the QC limits but MS/MSD data are outside QC limit.

1. MS/MSD Recoveries and Precision Criteria

The laboratory should use one MS and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If target analytes are not expected, MS/MSD should be analyzed.

NOTES: Data for MS and MSDs will not be present unless requested by the Region.
 Notify the Contract Laboratory COR if a field or trip blank was used for the MS and MSD.

For a Matrix Spike that does not meet criteria, apply the action to only the field sample used to prepare the Matrix Spike sample. If it is clearly stated in the data validation materials that the samples were taken through incremental sampling or some other method guaranteeing the homogeneity of the sample group, then the entire sample group may be qualified.

List the %Rs, RPD of the compounds which do not meet the criteria.

Sample ID: _____JC25442-2_____

Matrix/Level: _____Groundwater_____

The QC reported here applies to the following samples:
 JC25442-1, JC25442-2, JC25442-3, JC25442-4

Method: **SW846 8270D BY SIM**

| Compound | JC25442-2 ug/l | Q | Spike ug/l | MS ug/l | MS % | Spike ug/l | MSD ug/l | MSD % | RPD | Limits Rec/RPD |
|-------------|-------------------|---|---------------|------------|---------|---------------|-------------|----------|-----|-------------------|
| Naphthalene | ND a | | 2.04 | 1.54 | 75 | 2.04 | 1.59 | 78 | 3 | 23-140/36 |
| 1,4-Dioxane | 49.8 | | 2.04 | 44.8 | 0* b | 2.04 | 42.7 | 0* b | 5 | 20-160/30 |

(a) Result is from Run #2.

(b) Outside control limits due to high level in sample relative to spike amount.

Note: MS/MSD % recoveries and RPD within laboratory control limits except in the cases described in this document. No action taken, 1,4-dioxane outside control limits due to high level in sample relative to spike amount.

- * QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- * If QC limits are not available, use limits of 70 – 130 %.

DATA REVIEW WORKSHEETS

Actions:

| QUALITY | %R < LL | %R > UL |
|--------------------|---------|---------|
| Positive results | J | J |
| Nondetects results | R | Accept |

MS/MSD criteria apply only to the unspiked sample, its dilutions, and the associated MS/MSD samples:

If the % R for the affected compounds were < LL (or 70 %), qualify positive results (J) and nondetects (UJ).

If the % R for the affected compounds were > UL (or 130 %), only qualify positive results (J).

If 25 % or more of all MS/MSD %R were < LL (or 70 %) or if two or more MS/MSD %Rs were < 10%, qualify all positive results (J) and reject nondetects (R).

A separate worksheet should be used for each MS/MSD pair.

DATA REVIEW WORKSHEETS

All criteria were met X
Criteria were not met
and/or see below

INTERNAL STANDARD PERFORMANCE

The assessment of the internal standard (IS) parameter is used to assist the data reviewer in determining the condition of the analytical instrumentation.

List the internal standard area of samples which do not meet the criteria.

| DATE | SAMPLE ID | IS OUT | IS AREA | ACCEPTABLE RANGE | ACTION |
|------|-----------|--------|---------|---------------------|--------|
|------|-----------|--------|---------|---------------------|--------|

Internal area meets the required criteria of batch samples corresponding to this data package.

Action:

1. If an internal standard area count for a sample or blank is greater than 213.0% of the area for the associated standard (opening CCV or mid-point standard from initial calibration) (see Table 10 below):
 - a. Qualify detects for compounds quantitated using that internal standard as estimated low (J-).
 - b. Do not qualify non-detected associated compounds.
2. If an internal standard area count for a sample or blank is less than 20.0% of the area for the associated standard (opening CCV or mid-point standard from initial calibration):
 - a. Qualify detects for compounds quantitated using that internal standard as estimated high (J+).
 - b. Qualify non-detected associated compounds as unusable (R).
3. If an internal standard area count for a sample or blank is greater than or equal to 50.0%, and less than or equal to 213% of the area for the associated standard opening CCV or mid-point standard from initial calibration, no qualification of the data is necessary.
4. If an internal standard RT varies by more than 10.0 seconds: Examine the chromatographic profile for that sample to determine if any false positives or negatives exist. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for that sample fraction. Detects should not need to be qualified as unusable (R) if the mass spectral criteria are met.
5. If an internal standard RT varies by less than or equal to 10.0 seconds, no qualification of the data is necessary.

DATA REVIEW WORKSHEETS

Note: Inform the Contract Laboratory Program Project Officer (CLP PO) if the internal standard performance criteria are grossly exceeded. Note in the Data Review Narrative potential effects on the data resulting from unacceptable internal standard performance.

State in the Data Review Narrative if the required internal standard compounds are not added to a sample or blank or if the required internal standard compound is not analyzed at the specified concentration.

Actions:

Table 10. Internal Standard Actions for Semivolatile Analysis

| Criteria | Action | |
|--|------------------|------------------|
| | Detect | Non-detect |
| Area response < 20% of the opening CCV or mid-point standard CS3 from ICAL | J+ | R |
| 20% ≤ Area response < 50% of the opening CCV or mid-point standard CS3 from ICAL | J+ | UJ |
| 50% ≤ Area response ≤ 200% of the opening CCV or mid-point standard CS3 from ICAL | No qualification | No qualification |
| Area response > 200% of the opening CCV or mid-point standard CS3 from ICAL | J- | No qualification |
| RT shift between sample/blank and opening CCV or mid-point standard CS3 from ICAL > 10.0 seconds | R | R |
| RT shift between sample/blank and opening CCV or mid-point standard CS3 from ICAL < 10.0 seconds | No qualification | No qualification |

DATA REVIEW WORKSHEETS

All criteria were met X
 Criteria were not met
 and/or see below

TARGET COMPOUND IDENTIFICATION

Criteria:

Is the Relative Retention Times (RRTs) of reported compounds within ± 0.06 RRT units of the standard RRT [opening Continuing Calibration Verification (CCV) or mid-point standard from the initial calibration].
Yes? or No?

List compounds not meeting the criteria described above:

| Sample ID | Compounds | Actions |
|-----------|-----------|---------|
| ===== | ===== | ===== |
| _____ | _____ | _____ |
| _____ | _____ | _____ |
| _____ | _____ | _____ |

Mass spectra of the sample compound and a current laboratory-generated standard [i.e., the mass spectrum from the associated calibration standard (opening CCV or mid-point standard from initial calibration)] must match according to the following criteria:

- All ions present in the standard mass spectrum at a relative intensity greater than 10% must be present in the sample spectrum.
- The relative intensities of these ions must agree within $\pm 20\%$ between the standard and sample spectra (e.g., for an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30-70%).
- Ions present at greater than 10% in the sample mass spectrum, but not present in the standard spectrum, must be evaluated by a reviewer experienced in mass spectral interpretation.

List compounds not meeting the criteria described above:

| Sample ID | Compounds | Actions |
|-----------|-----------|---------|
| ===== | ===== | ===== |
| _____ | _____ | _____ |
| _____ | _____ | _____ |
| _____ | _____ | _____ |

Identified compounds meet the required criteria

DATA REVIEW WORKSHEETS

Action:

1. The application of qualitative criteria for GC/MS analysis of target compounds requires professional judgment. It is up to the reviewer's discretion to obtain additional information from the laboratory. If it is determined that incorrect identifications were made, qualify all such data as unusable (R).
2. Use professional judgment to qualify the data if it is determined that cross-contamination has occurred.
3. Note in the Data Review Narrative any changes made to the reported compounds or concerns regarding target compound identifications. Note, for Contract Laboratory COR action, the necessity for numerous or significant changes.

TENTATIVELY IDENTIFIED COMPOUNDS (TICS)

NOTE: Tentatively identified compounds should only be evaluated when requested by a party from outside of the Hazardous Waste Support Section (HWSS).

List TICs

| Sample ID | Compound | Sample ID | Compound |
|-----------|----------|-----------|----------|
| ===== | | | |
| _____ | | _____ | |
| _____ | | _____ | |
| _____ | | _____ | |
| _____ | | _____ | |

Action:

1. Qualify all TIC results for which there is presumptive evidence of a match (e.g. greater than or equal to 85% match) as tentatively identified (NJ), with approximated concentrations. TICs labeled "unknown" are qualified as estimated (J).
2. General actions related to the review of TIC results are as follows:
 - a. If it is determined that a tentative identification of a non-target compound is unacceptable, change the tentative identification to "unknown" or another appropriate identification, and qualify the result as estimated (J).
 - b. If all contractually-required peaks were not library searched and quantitated, the Region's designated representative may request these data from the laboratory.
3. In deciding whether a library search result for a TIC represents a reasonable identification, use professional judgment. If there is more than one possible match, report the result as "either compound X or compound Y". If there is a lack of isomer specificity, change the TIC result to a nonspecific isomer result (e.g., 1,3,5-trimethyl benzene to trimethyl benzene isomer) or to a compound class (e.g., 2-methyl, 3-ethyl benzene to a substituted aromatic compound).
4. The reviewer may elect to report all similar compounds as a total (e.g., all alkanes may be summarized and reported as total hydrocarbons).

DATA REVIEW WORKSHEETS

5. Target compounds from other fractions and suspected laboratory contaminants should be marked as "non-reportable".
6. Other Case factors may influence TIC judgments. If a sample TIC match is poor, but other samples have a TIC with a valid library match, similar RRT, and the same ions, infer identification information from the other sample TIC results.
7. Note in the Data Review Narrative any changes made to the reported data or any concerns regarding TIC identifications.
8. Note, for Contract Laboratory COR action, failure to properly evaluate and report TICs

DATA REVIEW WORKSHEETS

All criteria were met X
 Criteria were not met
 and/or see below

SAMPLE QUANTITATION AND REPORTED CONTRACT REQUIRED QUANTITATION LIMITS (CRQLS)

Action:

1. When a sample is analyzed at more than one dilution, the lower CRQL are used unless a QC exceedance dictates the use of higher CRQLs from the diluted sample. Samples reported with an "E" qualifier should be reported from the diluted sample.
2. If any discrepancies are found, the Region's designated representative may contact the laboratory to obtain additional information that could resolve any differences. If a discrepancy remains unresolved, the reviewer must use professional judgment to decide which value is the most accurate. Under these circumstances, the reviewer may determine that qualification of data is warranted. Note in the Data Review Narrative a description of the reasons for data qualification and the qualification that is applied to the data.
3. For non-aqueous samples, if the solids is less than 10.0%, use professional judgment for both detects and non-detects. If the percent solid for a soil sample is greater than or equal to 10.0% and less than 30.0%, use professional judgment to qualify detects and non-detects. If the percent solid for a soil sample is greater than or equal to 30.0%, detects and non-detects should not be qualified (see Table 11).
4. Note, for Contract Laboratory COR action, numerous or significant failures to accurately quantify the target compounds or to properly evaluate and adjust CRQLs.
5. Results between MDL and CRQL should be qualified as estimated "J".
6. Results < MDL should be reported at the CRQL and qualified "U". MDLs themselves should not be reported.

Table 11. Percent Solids Actions for Semivolatile Analysis for Non-Aqueous Samples

| Criteria | Action | |
|-------------------------|---------------------------|---------------------------|
| | Detects | Non-detects |
| %Solids < 10.0% | Use professional judgment | Use professional judgment |
| 10.0% ≤ %Solids ≤ 30.0% | Use professional judgment | Use professional judgment |
| %Solids > 30.0% | No qualification | No qualification |

SAMPLE QUANTITATION

The sample quantitation evaluation is to verify laboratory quantitation results. In the space below, please show a minimum of one sample calculation:

Sample ID: JC25442-2_MS_(SIM) Analyte: Naphthalene RF: 2.470

$$\begin{aligned}
 [] &= (71830)(4.0)/(153856)(2.470) \\
 &= 0.76 \text{ ppm} \quad \text{Ok}
 \end{aligned}$$

DATA REVIEW WORKSHEETS

QUANTITATION LIMITS

A. Dilution performed

| SAMPLE ID | DILUTION FACTOR | REASON FOR DILUTION |
|-----------|-----------------|---------------------|
| | | |
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DATA REVIEW WORKSHEETS

All criteria were met _____
 Criteria were not met _____
 and/or see below _____ N/A _____

FIELD DUPLICATE PRECISION

Sample IDs: _____ - _____

Matrix: _____ - _____

Field duplicate samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

The project QAPP should be reviewed for project-specific information.

Suggested criteria: if large RPD (> 50 %) is observed, confirm identification of the samples and note differences. If both samples and duplicate are <5 SQL, the RPD criteria is doubled.

| COMPOUND | SQL ug/L | SAMPLE CONC. | DUPLICATE CONC. | RPD | ACTION |
|--|-------------|-----------------|--------------------|-----|--------|
| | | | | | |
| | | | | | |
| No field/laboratory duplicate analyzed as part of this data package. MS/MSD % recovery RPD used to assess precision. RPD within the required guidance document criteria < 50 % for detected target analytes above 5 SQL. | | | | | |
| | | | | | |
| | | | | | |

DATA REVIEW WORKSHEETS

All criteria were met X
 Criteria were not met
 and/or see below

OTHER ISSUES

A. System Performance

List samples qualified based on the degradation of system performance during sample analysis:

| Sample ID | Comments | Actions |
|-----------|----------|---------|
| ===== | ===== | ===== |
| _____ | _____ | _____ |
| _____ | _____ | _____ |
| _____ | _____ | _____ |
| _____ | _____ | _____ |

Action:

Use professional judgment to qualify the data if it is determined that system performance has degraded during sample analyses. Inform the Contract Laboratory Program COR any action as a result of degradation of system performance which significantly affected the data.

B. Overall Assessment of Data

List samples qualified based on other issues:

| Sample ID | Comments | Actions |
|-----------|----------|---------|
| ===== | ===== | ===== |
| _____ | _____ | _____ |
| _____ | _____ | _____ |
| _____ | _____ | _____ |
| _____ | _____ | _____ |

Note:

Action:

1. Use professional judgment to determine if there is any need to qualify data which were not qualified based on the Quality Control (QC) criteria previously discussed.
2. Write a brief narrative to give the user an indication of the analytical limitations of the data. Inform the Contract Laboratory COR the action, any inconsistency of the data with the Sample Delivery Group (SDG) Narrative. If sufficient information on the intended use and required quality of the data is available, the reviewer should include their assessment of the usability of the data within the given context. This may be used as part of a formal Data Quality Assessment (DQA).

DATA REVIEW WORKSHEETS

3. Sometimes, due to dilutions, re-analysis or SIM/Scan runs are being performed, there will be multiple results for a single analyte from a single sample. The following criteria and professional judgment are used to determine which result should be reported:
- The analysis with the lower CRQL
 - The analysis with the better QC results
 - The analysis with the higher results